Operation of a Reverse Osmosis Plant for the Partial Concentration of Maple Sap

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SUMMARY

A reverse osmosis unit containing 800 square feet of cellulose acetate membrane in the spirally wound module type has been constructed for the partial concentration of maple sap. Under a pressure of 600 psig, maple sap pumped through the unit at 5 gal/min loses 50% of its original volume of water, thus doubling its sugar content. This concentrated sap, when reduced to maple syrup by a conventional atmosphere boiling procedure, had the full flavor of maple syrup and was free of off-flavors.

INTRODUCTION

The manufacture of standard density (65.5° Brix) maple syrup requires that maple sap, which is a dilute sugar solution of 1° to 5° Brix, be concentrated 30- to 40-fold. This concentration is normally done by thermal distillation at atmospheric pressure in open evaporator pans. The thermal energy required for the processing, whether from wood or oil fuel, costs approximately 50 cents to produce one gallon of syrup.

Mangan *et al.* (1965) directed attention to the use of the reverse osmosis process for obtaining potable water from sea water. Later, Morgan *et al.* (1965) applied this principle of concentration to the removal of water from fruit juices. McDonough (1968) has applied it to the concentration of whey. Willits *et al.* (1967) demonstrated on a laboratory scale that the reverse osmosis process could be effectively used to concentrate maple sap. They pointed out that maple sap was different from other natural juices, being a single phase system with a solids content in the order of sea water, and like sea water it is ideally suited to concentration by reverse osmosis.

The laboratory scale experiments indicated that (1) the maple flavor precursors did not pass through the semipermeable membrane but were retained unchanged in the sap concentrate, (2) up to 75% of the sap water could be efficiently removed at an estimated energy cost of about 1/20 of that for thermal distillation, and (3) the removal of the remainder of the water can be done by the conventional heat process to produce maple flavor. The thermal distillation, boiling the sap in open pans at atmospheric pressure, provides the heat required for formation of the maple flavor from its precursors that are normal maple sap constituents.

Based upon these observations and data, a semi-plant-size reverse osmosis concentrator (Fig. 1) was constructed. A description of the unit and its instrumentation is being prepared. Hereafter this plant will be designated by the term EUROC (Eastern Utilization Reverse Osmosis Concentrator). Following extensive laboratory tests the EUROC was put into field operation during the 1968 sap flow season at a central maple sap evaporation plant. The main objectives of the field studies were to (1) determine the effect on the quality of syrup obtained from sap partially concentrated by reverse osmosis, (2) observe the performance of the EUROC with maple sap for prolonged periods of continuous operation with uniform feed material, and (3) determine the effect of high bacterial, yeast, and mold counts in the feed material (sap) on the performance of the EUROC when in continuous operation and after periods of shut-down. The results of the other studies are reported here.

Fig. 1. The EUROC, a reverse osmosis concentrator containing 800 square feet of membrane in spirally wound form.

EXPERIMENTAL

The EUROC is 12 ft long, 4 ft wide, and 5 ft high with 8 pressure vessels (each 10 ft long by 4 inches diameter), each containing 9 standard spirally wound reverse osmosis modules. Each module contains approximately 11 ft^2 of membrane surface. This provides a total of 800 ft^2 of modified cellulose acetate membrane. The modules are manufactured by General Atomic Division, Gulf Oil Corporation. The sap was pumped through the EUROC with a Moyno pump, which provided a continuous flow of sap to the modules at the desired pressures and flow rate.

The unit, designed primarily for research studies, is equipped with automated devices for flow control and for electronic data recording. In addition, sampling ports are so located that samples can be taken at any stage of the concentration. The 8 pressure vessels are so arranged that they can be operated either in parallel or series.

Upon completion of the construction of the EUROC, tap water was pumped through it to test for physical leaks and mechanical operation. Then a series of tests were made using a synthetic maple sap (2–3% sugar in water solution). This sugar solution was passed through the unit, with the pressure vessels operating in parallel, at different pressures and feed rates to determine the balance of these two factors that would produce a concentrated sap of the desired sugar content.

From the laboratory studies and data published by the manufacturer of the modules, flow rates of 4, 5, 6, and 7 gal/min, and pressures of 400, 500, and 600 psig were selected for study. The feed, concentrate, and by-product water were analyzed for Brix, temperature, specific conductivity, and rate of production. The "pure" water obtained is the by-product in the maple sap concentration and will, therefore, be termed "by-product water" in this paper. Also, the 8 pressure tubes were arranged to give 4 pairs of 2 tubes connected in series. The amount of water removed from the synthetic sap by both arrangements as a function of pressure and feed rate were determined.

The EUROC was then moved to the central maple sap evaporator plant of J. L. Sipple and Son, Bainbridge, New York, to be used for the purpose for which it was designed, the partial concentration of maple sap. The paramount factor that had to be established at the maple syrup plant was whether or not the concentrated sap obtained from the EUROC would yield a syrup having full-bodied maple flavor and free of off-flavors when concentrated to standard density syrup by use of the conventional open pan atmospheric evaporators. This was done by using 500 gal of sap taken from a uniformly mixed 4,000 gal of stored maple sap. The 500 gal were pumped through the EUROC pressure vessels at 600 psig at a feed rate of 5 gal/min. The treated sap was then boiled to syrup density in the commercial maple sap evaporator pans.

Following this, the EUROC was run for extended periods at 600 psig at several flow rates to test the uniformity of water removal. Also, the effect of higher pressures, 700 and 750 psig, on membrane flux and amount of concentration was determined. Again, for these tests the raw sap feed, the concentrate, and the by-product water were analyzed for degrees Brix, temperature, and specific conductivity. Also recorded were flow rates of the feed, by-product water, and concentrate.

RESULTS AND DISCUSSION

The data from the synthetic sap studies were used to construct the graphs in Fig. 2 and 3 to show the relationship of feed rate and pressure on the amounts of water removed from the feed (synthetic maple sap). In Fig. 2 it is shown that the general effect of pressure on the module membranes was according to theory, i.e. increasing the pressure caused increased membrane flux. Since the EUROC was designed to operate between 500 and 600 psig as the result of earlier work by the authors and recommendations of the manufacturers of the membrane modules and pressure tubes, no data were collected on pressure effects above 600 psig during the preliminary tests on the unit. Also, the results of earlier work led to the adjustment of the automatic feed controls to rates from 0–8 gal/min although the pump on the unit has a top capacity of 12 gal/min at low pressure. The data in Fig. 3 show the effect of changing the flow rate of the feed on the flux of the membranes. The curves indicate that flux is decreased at feed rates below 5 gal/min. A decrease in flux results for the lower operative pressure of 400 psig even at 5 gal/min. At 600 psig pressure the flux was essentially independent of feed rate.

To determine the optimum conditions of pressure and feed rate at which the EUROC should be operated to obtain the highest concentration of the sap and the removal of the greatest amount of by-product water per unit of time, the plot in Fig. 4 was constructed. By plotting the volume of by-product water obtained for each feed rate and operating pressure versus the percentage of water removed from the feed (raw sap), Fig. 4 shows

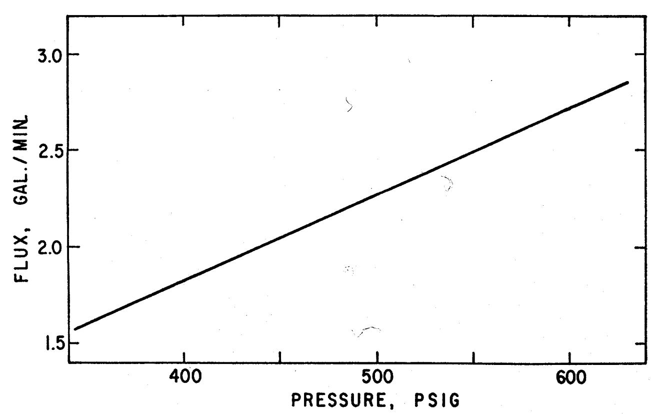

Fig. 2. The effect of increasing the working pressure on the flux of the membrane in EUROC.

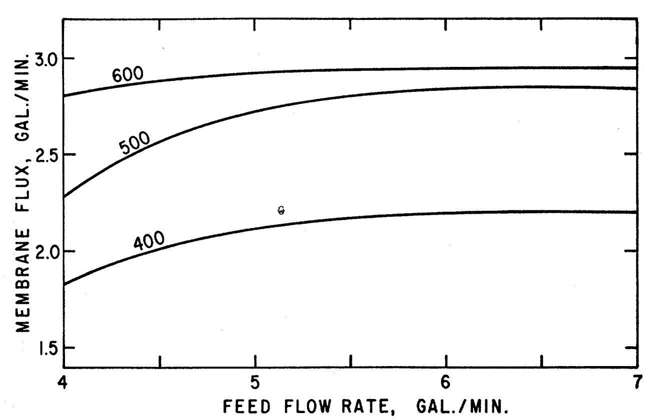

Fig. 3. The effect of increasing the feed rate to EUROC on the flux of the membrane at 400, 500, and 600 psig.

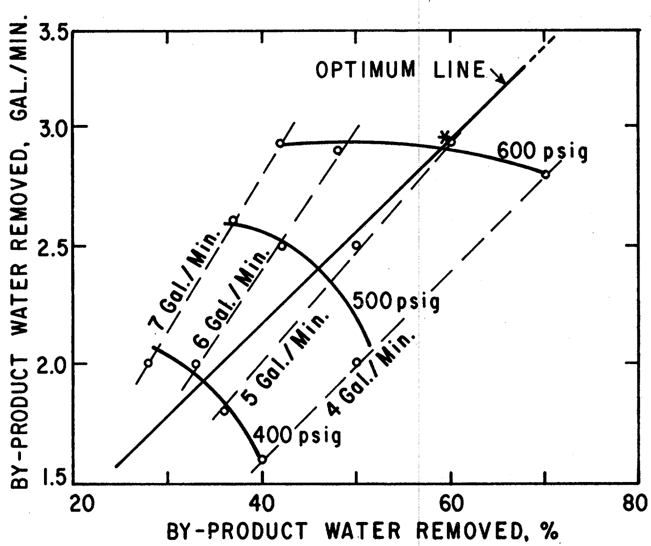

Fig. 4. A graph to show the operating conditions of feed rate and pressure for EUROC to obtain the optimum degree of concentrate relative to the amount of water removed.

Table 1. Comparison of flux obtained with pressure vessels in parallel and series.

Pressure psig	Feed rate gal/min	Membrane-flux gal/min	
		Parallel	Series
400	4	1.6	1.8
	5	1.8	1.9
	6	2.0	2.0
	7	2.0	—
	Avg.	1.85	1.9
500	4	2.0	2.2
	5	2.2	2.1
	6	2.5	2.1
	7	2.6	2.2
	Avg.	2.32	2.15
600	4	2.5	2.2
	5	2.7	2.7
	6	2.6	3.0
	7	2.6	—
	Avg.	2.6	2.6

that the optimum operating conditions are a feed rate of 5 gal/min at 600 psig. The measured membrane "flux" under these conditions, defined by the generally accepted definition as the gallons of water passing through one square foot of membrane surface per day, was 5.3 gal.

Table 1 shows that for EUROC there is but little difference in the amounts of by-product water obtained whether the pressure vessels are all arranged in parallel or in four pairs of two in series. This is because the flow rate of the feed to EUROC is regulated only for the unit as a whole. No means of individually controlling the flow rate in each pressure vessel was installed. The only advantage gained from the series arrangement is that it permits lowering the feed rate to the unit to one-half that permissible by the parallel arrangement. The velocity of flow across the membranes would then be the same as in the parallel arrangement. Since the flux is constant, lowering the feed rate results in an increase in the Brix of the sap concentrate, which is desirable. However, this advantage is nullified by the halving of the total capacity of the EUROC.

Field studies. The 500-gal portion of the 4,000 gal of stored sap (3.1° Brix) was partially concentrated by the EUROC to 5.5° Brix. Thus 43% of the water had been removed. This then was concentrated to standard density syrup by atmospheric boiling to produce a syrup of excellent quality. The flavor was full-bodied and was indistinguishable from that of syrup from the same lot of stored sap made entirely by evaporating the sap in the conventional open pan evaporators. This indicated that none of the maple flavor precursors in the sap had been lost by passage through the semipermeable membrane. Also, no foreign or "off" flavors were imparted to the sap by the membrane modules while being concentrated by the EUROC. Further, the by-product water exhibited no color or flavor on evaporation to dryness.

Having established that the EUROC had no effect on the quality of the syrup obtained, the unit was operated the remainder of the abnormally short maple sap flow season to determine flux characteristics as influenced by operating pressure and feed rates at extended periods of continuous operation, and to observe effects of and the control of microbial growth in the EUROC system. Ten thousand gal of sap were processed by the EUROC and the partially concentrated sap was discharged to the feed line of the open pan evaporators of the Sipple plant where it was evaporated, by boiling, to standard density syrup. At no time was there evidence in the finished syrup of any deleterious effect of the EUROC processing.

Effect of temperature on flux. Comparison of the observed data on the maple sap processed by the EUROC with the data on the sugar water studies indicated that the unit had a lower water flux with the maple sap than with sugar water. At first this discrepancy was thought to be due to some difference in the two sugar solutions. However, this was not the case as it was found that these differences in flux were due to the temperatures of the feed solutions (sugar water or sap) in contact with the membrane modules. A 10°F rise in temperature caused a 20–25% increase in the flux. Since the temperature could not be controlled, the data to be compared were normalized to 60°F.

Analysis of the water removed. The concentration of solids in the "by-product water" was continuously monitored by a specific conductivity meter, whose cell was mounted in the by-product water stream. The specific conductance varied between 28 and 40 micromhos indicating a very good rejection of electrolytes from the feed, which had specific conductance of 360 for sugar water and 275 for maple sap, respectively.

The by-product waters were sparkling clear and water white. On evaporation to dryness by boiling at atmospheric pressure, no odor was detectable in the steam and only a colorless microscopic film was produced on the surface of the evaporator vessel.

The analyses of the by-product water from several different runs are shown in Table 2. The samples contained from 380 to 1510 ppm total solids, 15 to 19 ppm sulfate ash, and 56 to 88 ppm total sugars as invert

Table 2. Analysis of by-product water from maple sap.

Sample No.	S-5	S-18	S-29
Sp. conductance (micromhos)	26	37	37
Total solids, ppm	1510	380	830
Ash, ppm	19	15	20
Sugar, ppm	79	88	56

Table 3. Energy costs of concentration of maple sap.

		Cents
Removal of 1 gal of water		
By reverse osmosis (electricity)		0.06
By thermal distillation (oil fuel)		1.50
Concentration of 2.5° Brix sap to 1 gal of syrup		
Thermal distillation (oil fuel)		49.8
Combination—		
55% Reverse osmosis	1.1	23.5
45% Distillation	22.4	
Savings by partial use of reverse osmosis—54%		

sugar. The amounts of sugar lost from the sap through the semipermeable membrane account for a loss of only one part in 500 of the solids in the raw sap.

The permeation of non-sugar solids (predominantly the organic acid salts of calcium and magnesium, commonly known in the maple syrup industry as "sugar sand") into the by-product water was also very small. The removal of even this small amount of sugar sand by permeation through the membranes into the by-product water is desirable since it causes a reduction in the amount of "sugar sand" precipitated in maple syrup manufacture that must be removed by filtration. It is hoped that reverse osmosis membranes will be developed that will permit removal of even larger amounts of sugar sand.

Energy costs. No data are available on the life expectancy of the reverse osmosis membranes nor on the cost of a commercially produced EUROC. Therefore, the cost data will have to be limited at the present time to the energy cost of removal of water from maple sap by reverse osmosis versus that for removal by thermal distillation using conventional atmospheric sap evaporators.

The energy costs to remove the large volumes of water from maple sap to produce syrup is an important cost item in syrup manufacture. Starting with 2.5° Brix sap, 33 gal of water must be removed to concentrate it to 1 gal of syrup. Table 3 shows the comparative energy costs to remove this water by the conventional thermal distillation (boiling in open pans) and by reverse osmosis. The thermal distillation requires 3.3 gal of No. 2 fuel oil to heat the sap to boiling and convert it to steam. The cost of the oil at 15 cents/gal is 49.5 cents or 1.5 cents/gal of syrup produced.

Operating the EUROC at the optimum pressure of 600 psig and a sap feed rate of 300 gal/hr removed 168 gal of water per hr from the sap. The hourly power consumption of the 7-h.p. motor used to drive the pump of the EUROC was 6.95 kw and the hourly energy cost at 1.5 cents per kw was 10.4 cents. This is the energy cost for removing the 168 gal of water or 0.062 cents per gal. This is 1/25 that of the comparable oil cost.

In the 1968 trials the EUROC removed an average of 55% of the water, at a cost of 1.1 cents, and the remaining water was removed by boiling, using No. 2 fuel oil at a cost of 22.4 cents. The total energy cost of this combination of water removal methods is 23.5 cents. This produced a savings of 54% in energy costs.

Sanitation and the EUROC. One of the important factors that will determine the economic feasibility of reverse osmosis is the life of the membrane. To protect the membranes in the EUROC from deterioration by infection from microorganisms normally in the raw sap and to prevent build-up of bacteria, yeasts or molds in the various lines and pressure vessels of the unit during extended use, the feed line is equipped with in-line ultraviolet water sterilizing units. Also, the unit is sanitized at the beginning and end of each use period with a solution of dilute hypochlorite. The excess chlorine is removed from the solution in the pressure vessels by flushing with water sterilized by passing it through the ultraviolet feed line purifier. Details of the sanitizing studies are being reported in detail in another paper.

The field tests of the EUROC for concentrating maple sap under commercial operating conditions were completely successful. They demonstrated that the operating cost of sap concentration can be effectively reduced by using reverse osmosis as part of the process. Even more important, they showed that removal of water from sap by reverse osmosis had no effect on the quality of the syrup subsequently obtained. The flavor was identical to that of syrup made by conventional thermal distillation.

The membrane modules used were of a new design with improved rejection and mechanical properties but a lower flux rate than the membranes used in the laboratory studies. However, still newer modules are now available that are reported to increase the flux by 100%. This would increase the capacity of the EUROC by a like amount with no change in any of its hardware or design. These new modules will be tested and reported in a later paper.

REFERENCES

Mangan, G. F., Jr. and Shackelford, J. M. 1965. In "Saline Water Conversion Report for 1964," pp. 30–39. U. S. Dept. Interior.

McDonough, F. E. 1968. Whey concentration by reverse osmosis. *Food Eng.* **40**(3), 124–27.

Morgan, A. I., Jr., Lowe, E., Merson, R. L. and Durkee, E. L. 1965. Reverse Osmosis. *Food Technol.* **19**, 1790–92.

Willits, C. O., Underwood, J. C. and Merten, U. 1967. Concentration by reverse osmosis of maple sap. *Food Technol.* **21**, 24–26.

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